

REC'D 19 JUL 2004	
WIPO	PCT



IB/2004/01921

Sertifikaat

REPUBLIEK VAN SUID AFRIKA

PATENT KANTOOR
DEPARTEMENT VAN HANDEL
EN NYWERHEID

Certificate

RÉPUBLIQUE OF SOUTH AFRICA

PATENT OFFICE
DEPARTMENT OF TRADE AND
INDUSTRY

Hiermee word gesertifiseer dat
This is to certify that

the documents attached hereto are true copies of Forms P1, P2
and provisional specification and drawing of South African Patent Application
No. 2003/4589 in the name of Element Six (Pty) Ltd

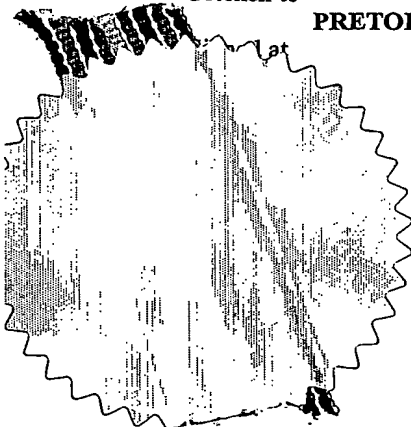
Filed : 12 June 2003 ✓
Entitled : Composition material
for Drilling Application

**PRIORITY
DOCUMENT**
SUBMITTED OR TRANSMITTED IN
COMPLIANCE WITH RULE 17.1(a) OR (b)

Geteken te
PRETORIA

in die Republiek van Suid-Afrika, hierdie
in the Republic of South Africa, this

23th dag van June 2004
day of



.....
Registrar of Patents

REPUBLIC OF SOUTH AFRICA		REGISTER OF PATENTS		PATENTS ACT, 1978		
OFFICIAL APPLICATION		LODGING DATE: PROVISIONAL		ACCEPTANCE DATE		
21	01 12003/4589	22	12 JUN 2003	47		
INTERNATIONAL CLASSIFICATION		LODGING DATE: COMPLETE		GRANTED DATE		
51		23				
FULL NAME(S) OF APPLICANT(S)/PATENTEE(S)						
71	ELEMENT SIX (PTY) LTD					
APPLICANTS SUBSTITUTED:				DATE REGISTERED		
71						
ASSIGNEE(S)				DATE REGISTERED		
71						
FULL NAME(S) OF INVENTOR(S)						
72	1. SIGALAS, IAKOVOS 2. MASETE, MOSIMANEGAPE STEPHEN					
PRIORITY CLAIMED		COUNTRY		NUMBER		
N.B. Use International abbreviation for country (see Schedule 4)		33	NIL	31	NIL	
				32	NIL	
TITLE OF INVENTION						
54	COMPOSITE MATERIAL FOR DRILLING APPLICATIONS					
ADDRESS OF APPLICANT(S)/PATENTEE(S)						
DEBID ROAD, NUFFIELD, SPRINGS, 1559, GAUTENG, SOUTH AFRICA						
ADDRESS FOR SERVICE				S & F REF		
74	SPOOR & FISHER, SANDTON			PA135283/P		
PATENT OF ADDITION NO.			DATE OF ANY CHANGE			
61						
FRESH APPLICATION BASED ON			DATE OF ANY CHANGE			

SPOOR & FISHER

REPUBLIC OF SOUTH AFRICA
PATENTS ACT, 1978
APPLICATION FOR A PATENT
AND ACKNOWLEDGEMENT OF RECEIPT
(Section 30 (1) - Regulation 22)

REPUBLIC OF SOUTH AFRICA
REVENUE FORM P.1

R 0060,00

HASH 711

The granting of a patent is hereby requested by the undermentioned applicant on the basis of the present application filed in duplicate

OFFICIAL APPLICATION NO.

21 01 2003/4589

REPUBLIC VAN SUID AFRIKA
S & F REFERENCE

PA135283/P

FULL NAME(S) OF APPLICANT(S)

71 ELEMENT SIX (PTY) LTD

ADDRESS(ES) OF APPLICANT(S)

DEBID ROAD, NUFFIELD, SPRINGS, 1559, GAUTENG, SOUTH AFRICA

TITLE OF INVENTION

54 COMPOSITE MATERIAL FOR DRILLING APPLICATIONS

THE APPLICANT CLAIMS PRIORITY AS SET OUT ON THE ACCOMPANYING FORM P.2. THE EARLIEST PRIORITY CLAIM IS:

COUNTRY: NIL NUMBER: NIL DATE: NIL

THIS APPLICATION IS FOR A PATENT OF ADDITION TO PATENT APPLICATION NO.

21 01

THIS APPLICATION IS A FRESH APPLICATION IN TERMS OF SECTION 37 AND IS BASED ON APPLICATION NO.

21 01

THIS APPLICATION IS ACCOMPANIED BY:

- ☒ 1. A single copy of a provisional specification of 10 pages.
- ☒ 2. Drawings of 1 sheet.
- ☐ 3. Publication particulars and abstract (Form P.8 in duplicate).
- ☐ 4. A copy of Figure of the drawings (if any) for the abstract.
- ☐ 5. Assignment of Invention.
- ☐ 6. Certified priority document.
- ☐ 7. Translation of the priority document.
- ☐ 8. Assignment of priority rights.
- ☐ 9. A copy of the Form P.2 and the specification of S.A. Patent Application No .
- ☐ 10. Declaration and power of attorney on Form P.3.
- ☐ 11. Request for ante-dating on Form P.4.
- ☐ 12. Request for classification on Form P.9.
- ☒ 13. Form P.2 in duplicate.
- ☐ 14. Other.

74 ADDRESS FOR SERVICE: SPOOR & FISHER, SANDTON

Dated: 12 June 2003

SPOOR & FISHER
PATENT ATTORNEYS FOR THE APPLICANT(S)

REGISTRAR OF PATENTS DESIGNS, TRADE MARKS AND COPYRIGHT
2003-06-12
REGISTRATEUR VAN PATENTE, MODELLE, F. INDELSMERKE EN ONTEURSREG
REGISTRAR OF PATENTS

REPUBLIC OF SOUTH AFRICA
PATENTS ACT, 1978**PROVISIONAL SPECIFICATION**

(Section 30(1) – Regulation 27)

OFFICIAL APPLICATION NO.

LODGING DATE

21	01	2003/ 4589
----	----	-------------------

22	
----	--

FULL NAMES OF APPLICANTS

71	ELEMENT SIX (PTY) LTD
----	-----------------------

FULL NAMES OF INVENTORS

72	SIGALAS, IAKOVOS MASETE, MOSIMANEGAPE STEPHEN
----	--

TITLE OF INVENTION

54	COMPOSITE MATERIAL FOR DRILLING APPLICATIONS
----	--

BACKGROUND OF THE INVENTION

This invention relates to a composite material, a method of making the composite material and a method of making a tool component.

Polycrystalline diamond (herein referred to as PCD) cutters are well-known and widely used in drill bit technology as the cutting element in drill bits used in core drilling, oil and gas drilling, and other similar applications. Such cutters generally comprise a PCD table formed on a hard metal substrate by a high temperature – high pressure sintering process. The substrate is then either brazed on an elongated support, or is directly brazed in a pocket of the drill bit, in a manner that exposes the PCD table to the surface for rock cutting.

It is known that PCD cutters with a thick PCD table can enjoy a prolonged useful life during the drilling operation. This is simply due to the fact that there is more of the ultrahard phase available for performing the rock cutting and shearing operation. However, a cutter with a thick PCD table layer, once a wear scar is formed, attacks the rock by applying the cutting loads on the rock through the part of the PCD table that is in contact with it. If the PCD layer is much thicker than those found in conventional cutters, then the cutting loads are distributed over a large area on the face of the rock being engaged. Therefore the cutting stresses are low, and the cutting efficiency is reduced. An engineering solution to maintaining a sharp edge is proposed by US 5,301,762 to Besson, in which proportioned grooves are placed in the structure, in order to generate a chipping effect that will generate a sharp fresh cutting edge. Such a technique uses up a significant proportion of the cutter's useful PCD material by fracturing it away, and cannot be cost effective.

In EP 0 196 777 to Wardley a way of maintaining sharpness of the PCD table by grading the wear resistance of said table is proposed. Specifically, the top layer is made of more wear resistant material than the layer adjacent to the substrate of the drilling cutter. The result is that the PCD layer below the top surface wears faster than the top layer, thus giving rise to a lip. This development generates a self-sharpening effect. The improved wear resistance is typically achieved by making the top PCD layer out of a fine grained PCD, and/or by reducing the amount of binder material used in comparison to the amount used to make less wear resistant layers. However, such thick PCD layers, when they contain fine grain diamond, are difficult to infiltrate with the necessary binder material. Therefore such cutters are difficult to sinter.

It is claimed in US 4,311,490 that such problems can be overcome by using coarse diamond particles as the starting material for the PCD layers adjacent to the substrate. Although such an approach helps, it does not eliminate the problem of infiltrating the top fine-grained layers, which in modern cutters can have substantial thickness. As such PCD tables become thicker, toughness of the PCD table becomes increasingly important, due to the volume effect associated with the fracture probability of brittle materials. A number of disclosures deal with this problem.

In US 6,361,873 to Siracki *et al* an ordered structure of two phases that enhances the material's chipping resistance is disclosed. This is a very general patent, and does not address any other aspect than toughness of a structure. In US 4,604,106 it is suggested that the admixing of two phases in a random, but graded manner can be used to address stress management, as well as toughening of the structure. US 6,06,352 to Rai and US 2002/194955 disclose coated fibre and granule structures, respectively, in order to generate tough structures. In both the fibres and the granules, the core is made from ultrahard polycrystalline based materials, while the rim is made from hardmetals or cermets. In all these

cases, these advantages are attained at the sacrifice of wear resistance, as a result of the introduction of the less wear resistant cermets.

SUMMARY OF THE INVENTION

According to one aspect of the invention, a composite material comprises a plurality of cores of ultra-hard material, or the components for making an ultra-hard material, dispersed in a matrix, the matrix comprising the components for making an ultra-hard material of a grade different to that of the cores and a suitable binder.

The ultra-hard material is polycrystalline in nature and is typically PCD or PCBN.

The cores are typically provided as individual particles or in the form of granules.

In one embodiment of the invention the cores are made from a fine-grained PCD grade material and the matrix of a coarser PCD grade material than that of the cores.

In an alternative embodiment of the invention, the cores may be formed of a coarser PCD grade material and the matrix of a fine-grained PCD grade material.

In a further alternative embodiment of the invention, the cores may be formed of PCD and the matrix of PcBN type material. Such PcBN may encompass cubic boron nitride grains sintered to themselves, and/or with any number of ceramic phases comprising nitrides, borides, carbides or carbonitrides of aluminium, titanium, tungsten, hafnium, zirconium, silicon, niobium, and tantalum.

In yet another alternative embodiment of the invention, the cores may be formed from PcBN type material. Such PcBN may encompass cubic boron nitride grains sintered to themselves, and/or with any number of ceramic phases comprising nitrides, borides, carbides or carbonitrides of aluminium, titanium, tungsten, hafnium, zirconium, silicon, niobium, and tantalum. In this embodiment the rim may be formed out of PCD.

The fine-grained PCD grade material preferably has grains having a grain size in the range 0.1 to 20 microns. The coarser PCD grade material preferably has grains having a grain size in the range 10 to 100 microns. If core and rim are made from the same ultrahard material, then the particle size of the core is significantly different from that of the rim, by between 5 and 70 micron. Alternatively, the core and the rim of the granules can both be made from the same material, but with different binder phases.

Alternatively, the core and the rim of the granules may be made from mixtures of two types of ultrahard materials, such as PCD and PcBN, but those mixtures being substantially different from each other.

According to a further aspect of the invention, a method of producing a composite material as described above includes the steps of:

- (i) Providing a plurality of cores of an ultra-hard material or the components for making an ultra-hard material;
- (ii) Providing the components for making an ultra-hard material of a different grade to that of the cores and a suitable binder; and
- (iii) Consolidating the cores, components and binder to produce a composite material.

According to yet another aspect of the invention, a method of producing a tool component includes the steps of:

- (i) Providing a substrate;
- (ii) Providing a composite material as described above;
- (iii) Placing a layer of the composite material on a surface of the substrate to produce an unbonded component; and
- (iv) Subjecting the unbonded component to conditions of elevated temperature and pressure suitable to produce an ultra-hard material.

The cores are typically provided as granules coated with the components for making an ultra-hard material and the binder.

The composite may be a moulded composite, which takes on the shape of the surface of the substrate on which it is placed. In this regard, the composite may be pre-cast in the appropriate form or, alternatively, moulded *in situ*.

The composite material typically takes on a honeycomb structure of an ultra-hard material and cores within the pores of the honeycomb structure bonded to the honeycomb structure. The pores of the honeycomb structure may be ordered or random.

The components necessary to produce either grade of ultra-hard material may comprise a mass of ultra-hard abrasive particles and optionally a second phase comprising a solvent/catalyst or a precursor to a solvent/catalyst, in particulate form, for the ultra-hard abrasive particle. Such components may include the superalloys, such as the Nimonic® and Stellite® alloys, and high temperature brazes as well as the metals iron, nickel and cobalt. They may also contain other elements such as titanium, tungsten, tantalum, silicon, molybdenum, zirconium, and niobium. The second phase may also be silicon carbide, this phase being generated by

admixing silicon or its precursors with the diamond grains prior to sintering, and by reaction-sintering the mix when sintering the whole body of the granules at high temperatures and pressures.

The sintering conditions are such that the ultra-hard abrasive particles are crystallographically stable.

The particles in the core and in the coating are provided in a suitable binder, such as an organic binder. This binder will preferably be removed prior to the sintering of step (iv). Examples of suitable binders include but are not limited to camphor, methylcellulose and polyethylene glycol.

The plurality of granules may be consolidated by applying pressure to the granules, for example, in a confined space such as a die. The consolidated composite is a green state product which has coherency, but which may also be severed, for example, by cutting. A piece which may be severed and removed from the consolidated or coherent composite has flexibility and may be applied to surfaces, which are flat or profiled, e.g. a curved surface. The die for consolidating the granules may be provided with one or both punches profiled such that the green state product has at least one surface that has a shape complementary to the substrate upon which it is to be placed. In this case, the composite layer presents a working surface or a cutting edge. Either surface may be profiled to accommodate a further layer such as an ultra-hard material layer or another layer of a composite material but of a different composition, for instance having cores of hardmetal coated with ultra-hard material or the components for making the ultra-hard material, to provide for a grading of properties. Several interlayers of composite material having different compositions may be provided.

Where the cores of the additional composite material are formed from carbide particles, these will typically be tungsten carbide particles, titanium carbide particles, tantalum carbide particles or molybdenum carbide

particles. The metal binder may be any metal binder known in the art such as iron, nickel, cobalt or an alloy containing one or more of these metals.

The substrate will typically be a cemented carbide substrate.

The granules may be produced by providing a core and then coating the core with an ultra-hard material in the presence of an organic binder. Coating may take place by fluidising the cores and spraying the coating thereon or by palletising in a pan. The granules may be made of more than one layer, each layer comprising PCD of different grain sizes, or different types of PcBN, or mixtures of both.

BRIEF DESCRIPTION OF THE DRAWINGS

The invention will now be described in more detail, by way of example only, with reference to the accompanying drawings in which:

Figure 1 is a cross section through a granule used in making a composite material of the invention; and

Figure 2 is a cross section through a consolidated composite material of the invention.

DESCRIPTION OF EMBODIMENTS

Referring to Figure 1, a granule 10 comprises a core 12 and a coating 14 substantially enclosing the core 12. The granule 10 illustrated is of uniform shape and spherical. The granule does not have to be of such uniform shape, nor need it be spherical. Other shapes are possible.

In this embodiment, the core 12 is made from a fine-grained PCD grade material, typically in the range of 0.1 to 12 microns in grain size. This grade is endowed with high wear resistance and high strength.

The coating 14 is made from a coarser PCD grade material than that of the core 12, typically 10 to 100 microns in grain size. This grade has lower strength and wear resistance than the core material. The coarser grade material will also maintain large size pores during the high-pressure compaction process, thus providing wide conduits for the molten binder during the infiltration process.

The core 12 and coating 14 are both comprised of ultra-hard abrasive particles such as diamond or cubic boron nitride and optionally a metal or precursor in particulate form. Such metal may be a solvent/catalyst or another metal, which will sinter under the applied conditions of temperature and pressure.

An organic binder such as methylcellulose is present in both the core 12 and the coating 14 and provides both the core 12 and the coating 14 and the granule 10 as a whole with coherency. Other non-limiting examples of the organic binder include camphor and polyethylene glycol.

Figure 2 shows the resulting microstructure 16 after compaction of a plurality of granules 10. This microstructure now has a set of wear resistant cores 18, the so-called fine-grained phase, and a continuous or coarse-grained phase 20 of coarse-grained PCD that is not as strong, and is less wear resistant, than the cores 18. The result is that when cutting rock, the continuous phase 20 will wear preferentially, thus causing the wear resistant cores 18 to slightly protrude. These protrusions will concentrate the cutting forces on small areas of the rock face, thus improving cutting efficiency. This is a self-sharpening effect.

Since the fine-grained phase has a thermal expansion coefficient that is higher than that of the coarse-grained one, the fine-grained phase will be in

tension in the final compact. Therefore, in an alternative embodiment, if the structure is inverted with the fine-grained phase placed at the rim of the granules, and the coarse-grained phase at their core, the resulting compact will have a continuous phase that is in tension. Such a phase will encourage any cracks that run through it to follow the path defined by the continuous phase, thus forcing it to take a tortuous path. This will increase the fracture toughness, and therefore the chip and spall resistance of this phase.

In yet a further embodiment, the composite material of the invention can be combined with another material, made from granules with a hardmetal core and a PCD rim, to generate a continuously graded structure. Such a structure would offer the advantages of thick PCD layers, self-sharpening, and stress management by way of grading.

The coarse-grained continuous phase will present the infiltrating metal from the substrate with large pores, thus allowing for easy infiltration. This will make sintering of thick PCD layers made from such a material much easier to sinter than conventional fine grained PCD, or graded layers where the layer adjacent to the hardmetal substrate is made from coarse grains, and the layer furthest from the substrate is made from fine grained diamond.

The final size of the cores may be between 5 and 300 microns. The volume fraction of the cores in the sintered structure may be between 10 and 90 percent.

The PCD composites of the invention increase the possibility of sintering PCD layers of large thickness whilst maintaining good wear resistance, and providing the cutter with a self-sharpening effect.

DATED THIS 12th DAY OF JUNE 2003



SPOOR & FISHER
APPLICANT'S PATENT ATTORNEYS

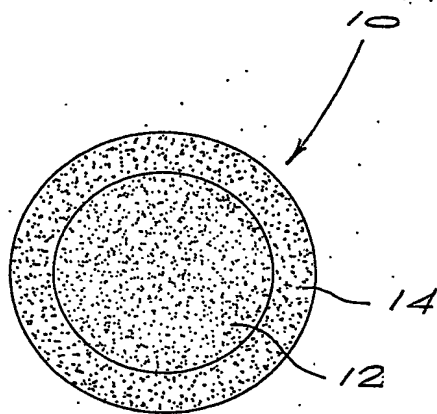


Fig. 1

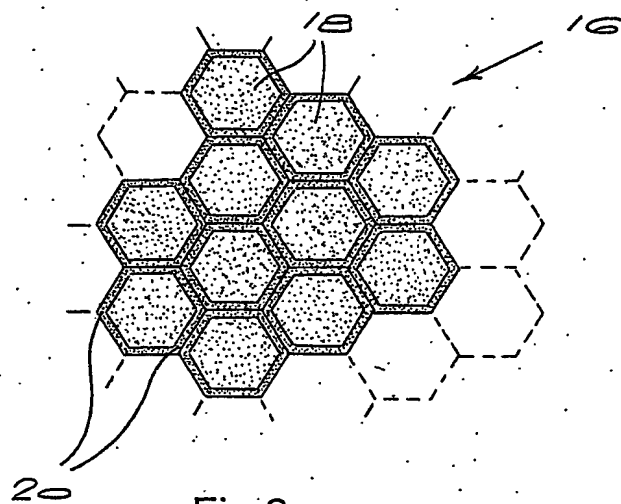


Fig. 2

[Handwritten signature]